

AFFIDAVIT



RECEIVED
NOV 14 2003
TECH CENTER 1600/2000

I, Dr. Hans Brühwiler, Patent Attorney, c/o Rottmann, Zimmermann + Partner AG, of 37 Glattalstrasse, CH-8052 Zurich (Switzerland), sincerely declare and depose as follows:

1. I am familiar with the German and English languages.
2. The annexed 14 pages are a complete and true English translation of the German text of the annexed Priority Document, issued by the Swiss Federal Institute for Intellectual Property (Swiss Patent Office) with respect to Swiss Patent Application No. 2000 2520/00, filed on 22 December 2000.

Declared at CH-8052 Zurich (Switzerland), this 9 October 2003.

Hans Brühwiler

Dr. Hans Brühwiler

(Blazon)

SWISS CONFEDERATION

Attestation

The annexed documents agree to the original technical documents of the Patent application for Switzerland and Liechtenstein specified on the next page. Switzerland and the Principality of Liechtenstein form a unitary territory of protection, Therefore, protection can be applied for only jointly for both countries.

Berne, 19 November 2001

(Ribbon and Official Seal)

Federal Institute for Intellectual Property

Patent Proceedings

(Signature)

Rolf Hofstetter

Patent Application No. 2000 2520/00

CERTIFICATE OF LODGMENT (Art. 46 Al. 5 Patent Rules)

The Federal Institute for Intellectual Property certifies the receipt of the Swiss Patent Application specified hereafter.

Title:

Cosmetic preparation, particularly hear treatment product or product for care of the mouth.

Applicant:

Mibelle AG Cosmetics
1 Bollimattstrasse
5033 Buchs

Agent:

Rottmann, Zimmermann + Partner AG
37 Glattalstrasse
8052 Zurich

Application Date: 22 December 2000

Presumable Classes: A61K

Cosmetic preparation, particularly hair treatment product or product for care of the mouth

The present invention relates to a cosmetic preparation as defined in claim 1, and particularly to a hair treatment product as defined in claim 4, and to a product for care of the mouth as defined in claim 10.

Antioxidants are essential substances in nature. They are used in cosmetics for protecting the compounds of said cosmetics and for protecting the skin against oxidative stress.

Oxidative stress is caused by ROS (Reactive Oxygen Species – reactive oxygen molecules). ROS are formed above all in water, since water is always saturated with oxygen.

Frequently, in cosmetics only oil soluble antioxidants are used, since quite a number of suitable substances are available, e.g. 3-tert.-butyl-4-hydroxyanisole or alpha-tocopherol.

The use of water soluble antioxidants is limited, since no suitable substances are available on the market.

Vitamin C (ascorbic acid) is a water soluble antioxidant, which is widespread in nature but is rarely used in cosmetics, since it is very unstable.

Oligomeric procyanidins, isolated from grape seeds or other plant parts, are very good antioxidants, which are useful in cosmetics. Procyanidins are polyphenols on the basis of catechin und epicatechin. The use of oligomeric procyanidins in combination with carotinoids was therefore also proposed for combating free radicals in foodstuffs, foodstuff complements as well as cosmetic and pharmaceutical preparations (US-A-5,648,377.

However, the use of oligomeric procyanidins is not unproblematic, since said compounds are only conditionally stable if dissolved in water. Therefore, so far the use

of such oligomeric procyanidins was essentially limited to dry products such as capsules or tablets as foodstuff complements.

Surprisingly it was now found that oligomeric procyanidins in an aqueous solution are stabilized by the addition of tocopherols, which are introduced into the aqueous solution by means of cosmetic solubilizers, e.g. PEG-40 hydrogenated castor oil. On the other hand, stability of the tocopherols is increased by the presence of oligomeric procyanidins.

Thus, e.g., the antioxidant activity of a mixture of 0.5 percent by weight of oligomeric procyanidins and 5 percent by weight of tocopherol remains stable for more than 8 months. Furthermore, this mixture of antioxidants also shows a synergistic effect with respect to the inhibition of UV-A induced formation of toxic substances in squalene.

It is known that hair is damaged by oxidative stress, e.g. by UV irradiation. However, damage of hair caused by heat drying, particularly by using an electric hair dryer, was scarcely investigated.

Surprisingly it was now found that this strain can provoke heavy damages, above all in view of the fact that treatments are carried out very often.

The damage is mainly caused by the activation of oxygen dissolved in the water film on the wet hair by the heat, that is that ROS (Reactive Oxygen Species – reactive oxygen molecules) may be formed. The ROS, e.g. singlet oxygen or oxygen radicals, then react with the constituents of the hair surface and thus damage the hair structure. Among other reactions, the proteins on the hair surface are oxidized and degraded. Said proteins may then be washed off from hair in the form of amino acids, peptides and proteins.

Therefore, one object of the present invention was to create a hair treatment product which protects hair against oxidative stress. Particularly, it should be based on an aqueous formulation and act both as a "leave on" product, i.e. a product remaining on hair, and as a "rinse off" product, i.e. a product which is washed out.

This object is achieved by the characteristics of claim 1 to 9.

The following tests prove in detail the surprising effects of the combination of active ingredients comprising oligomeric grape seed procyanidins and tocopherols. Particularly they show:

- Adsorption of tocopherol on the hair surface (Test 1);
- Protection of hair against damage on heat drying (Test 2); and
- Protection of hair against damage by sea water and UV irradiation (Test 3).

Further active ingredients of the hair treatment product of the present invention protecting hair against oxidation and/or degradation, particularly photo oxidation, are e.g.: vitamin C and vitamin C derivatives, vitamin A and vitamin A derivatives, perfume oils, unsaturated lipids and proteins.

In following tests and preparations the products are named according to the usual INCI declaration. All amounts are given in percents by weight.

Tests

The following tests were carried out with commercially available European hair and the following combination of active ingredients:

Ethyl Alcohol	30.0 %
Glycerin	40.0 %
PEG-40 Hydrogenated Castor Oil	10.0 %
Tocopherol	5.0 %
Procyanidins (Grape seed extract)	0.4 %
Water	ad 100 %

For the tests this combination of active ingredients was diluted with water to 2.5 %, said dilution of active ingredients being hereafter shortly called "active ingredients dilution".

Test 1 (Adsorption of tocopherol on the hair surface)

2 g of hair were incubated in 20 ml of the active ingredients dilution with stirring for 10, 20, and 60 minutes, respectively. Thereafter, the hair was rinsed with water and air dried. Then, the hair was extracted with 50 ml isopropanol each. The extracts were then vacuum dried and thereafter dissolved in 1 ml ethanol. The tocopherol content was determined by HPLC analysis.

Result

Incubation time	Amount of tocopherol on 2 g of hair
10 minutes	0.1 mg
20 minutes	0.4 mg
60 minutes	1.0 mg

Test 2 (Protection of hair against damage on heat drying)

The hair was first washed with a shampoo and rinsed. Then, the hair was incubated for 10 minutes in the active ingredients dilution. Thereafter it was rinsed with water and dried by means of an electric hair drier. The dried hair was again sprayed with water and again dried by means of an electric hair drier. This procedure was repeated 4 and 9 times, respectively. The hair was then extracted with a 2% aqueous sodium dodecylsulfate solution. The extract was then filtered. The protein and peptide content

of the extract was then determined by means of the method of Bradford. In the control the hair was treated the same way, except that the hair was incubated in water instead of in the active ingredients dilution.

Result: Amount of protein in the extract

After electric heat drying for 5 times

Control	30 µg/ml
Active ingredients combination	7 µg/ml

After electric heat drying for 10 times

Control	47 µg/ml
Active ingredients combination	19 µg/ml

Test 3 (Protection of hair against damage by sea water and UV irradiation)

The hair was first washed with a shampoo and rinsed. Then, the hair was incubated for 60 minutes in the active ingredients dilution. Thereafter it was rinsed with water and air dried. The dried hair was then irradiated with UV light (control: without UV irradiation). The hair was then extracted with a 2% aqueous sodium dodecylsulfate solution or with sea water. The extract was then filtered. The protein and peptide content of the extract was then determined by means of the method of Bradford. In the control the hair was treated the same way, except that the hair was incubated in water instead of in the active ingredients dilution.

Result: Amount of protein in the various extracts

Sodium Dodecyl Sulfate extract

Without UV, without active ingredients dilution	36 µg/ml
Without UV, with active ingredients dilution	10 µg/ml
With UV, without active ingredients dilution	50 µg/ml
With UV, with active ingredients dilution	12 µg/ml

Sea water extract

Without UV, without active ingredients dilution	16 µg/ml
Without UV, with active ingredients dilution	10 µg/ml
With UV, without active ingredients dilution	28 µg/ml

With UV, with active ingredients dilution

16 µg/ml

Preparations

A) Hair shampoo with grape seed procyanidins and tocopherol

Sodium Laureth Sulfate 70 %	12.00 %
Cocamido Betain 35 %	7.00 %
Perfume	0.50 %
Glycerin	0.02 %
Ethyl Alcohol	0.02 %
PEG-40 Hydrogenated Castor Oil	0.01 %
Preservative	0.1-1.0 %
Tocopherol	0.005 %
Procyanidins (Grape seed extract)	0.0005 %
Water	ad 100 %

B) Hair conditioner with grape seed procyanidins and tocopherol

Cetaryl Alcohol	4.50 %
Cetrimonium Chloride	2.50 %
Dimethicone Copolyol	5.00 %
Perfume	0.50 %
Citric Acid	for pH 3.5
Glycerin	2.0 %
Ethyl Alcohol	1.0 %
PEG-40 Hydrogenated Castor Oil	0.5 %
Tocopherol	0.25 %
Procyanidins (Grape seed extract)	0.10 %
Preservative	0.1-1.0 %
Water	ad 100 %

C) Hair tip fluid with grape seed procyanidins and tocopherol

Dimethicone	25.00 %
Cyclomethicone	71.00 %
Dimethicone Copolyol	2.00 %
Perfume	1.00 %

Glycerin	0.2 %
Ethyl Alcohol	0.4 %
PEG-40 Hydrogenated Castor Oil	0.4 %
Tocopherol	0.05 %
Preservative	0.1-1.0 %
Procyanidins (Grape seed extract)	0.05 %

D. Hair spray aerosol with grape seed procyanidins and tocopherol

Acrylates Copolymer	3.00 %
2-Amino-methyl-propanol	0.70 %
Water	20.00 %
Glycerin	0.02 %
PEG-40 Hydrogenated Castor Oil	0.02 %
Tocopherol	0.001 %
Procyanidins (Grape seed extract)	0.0005 %
Dimethyl ether	40.00 %
Water	ad 100 %

E) Styling gel with grape seed procyanidins and tocopherol

Carbomer	1.00 %
Sodium Hydroxide solution 30 %	1.10 %
PVP/VA Copolymer	4.00 %
Glycerin	5.00 %
Ethyl Alcohol	2.0 %
PEG-40 Hydrogenated Castor Oil	1.0 %
Tocopherol	0.5 %
Procyanidins (Grape seed extract)	0.1 %
Preservative	0.1-1.0 %
Water	ad 100 %

F) Conditioner with grape seed procyanidins, tocopherol and retinyl palmitate

Cetearyl Alcohol	4.50 %
Cetrimonium Chloride	2.50 %
Dimethicone Copolyol	5.00 %
Perfume	0.50 %

Citric Acid	for pH 3.5
Ceteareth-20	1.00 %
Glycerin	2.0 %
Ethyl Alcohol	2.0 %
PEG-40 Hydrogenated Castor Oil	1.0 %
Tocopherol	0.001 %
Procyanidins (Grape seed extract)	0.5 %
Retinyl Palmitate	1.00 %
Preservative	0.1-1.0 %
Water	ad 100 %

G) Hair tonic with grape seed procyanidins, tocopherol and borage oil

Ethyl Alcohol	30.00 %
Borage Seed Oil	2.5 %
PEG-60 Hydrogenated Castor Oil	1.00 %
PEG-40 Hydrogenated Castor Oil	1.00 %
Perfume	0.50 %
Tocopherol	0.25 %
Procyanidins (Grape seed extract)	0.25 %
Preservative	0.1-1.0 %
Water	ad 100 %

H) Hair water for treating the scalp with grape seed procyanidins and tocopherol

Preservative	0.1...1.0 %
Perfume	0.5 %
Polysorbate-20	2.0 %
Glycerin	3.0 %
Sorbitol	8.0 %
Ethyl Alcohol	2.0 %
PEG-40 Hydrogenated Castor Oil	2.00 %
Tocopherol	1.0 %
Procyanidins (Grape seed extract)	0.1 %
Preservative	0.1-1.0 %
Water	ad 100 %

I) Non-aerosol hair spray with grape seed procyanidins and tocopherol

Acrylates Copolymer	3.00 %
2-Amino-methyl-propanol	0.70 %
Ethyl Alcohol	10.00 %
Glycerin	0.6 %
PEG-40 Hydrogenated Castor Oil	0.3 %
Tocopherol	0.1 %
Procyanidins (Grape seed extract)	0.2 %
Preservative	0.1-1.0 %
Water	ad 100 %

While the use of antioxidants in skin care products is widespread, the application in products for care of the mouth is still very rare.

Thus, another object of the present invention to create a product for care of the mouth which protects the gingiva and the oral mucosa against oxidative stress and inflammations. Particularly, it should be based on aqueous formulations and contain water soluble active ingredients.

This object is achieved by the characteristics of claims 1 to 3 and 10 to 15.

Examples of further active ingredients of the product for care of the mouth which can be protected against oxidation and/or degradation according to the present invention are: vitamin C and vitamin C derivatives, vitamin A and vitamin A derivatives, aromas, unsaturated lipids and enzymes.

Examples of products for care of the mouth according to the invention are:

- Toothpaste with grape seed procyanidins and tocopherol (0.1 %);
- Tooth-wash with grape seed procyanidins and tocopherol (5 %);
- Gingiva gel with grape seed procyanidins and tocopherol (1 %);
- Mouth-wash with grape seed procyanidins and tocopherol (10 %);
- Gingiva gel with retinyl palmitate and grape seed procyanidins and tocopherol (10 %);
- Anti-plaque toothpaste with grape seed procyanidins and tocopherol (1 %).

Claims

1. A cosmetic preparation characterized by a content of (a) water soluble oligomeric procyanidins and (b) oil soluble tocopherols.
2. The cosmetic preparation of claim 1 characterized by a content of (a) oligomeric procyanidins containing grape seed extract and (b) oil soluble natural tocopherols, particularly alpha-, beta-, gamma- and/or delta-tocopherols.
3. The cosmetic preparation of claim 1 or 2, characterized by a content of 0.00005 to 5 percent by weight of procyanidins (a) and of 0.00025 to 10 percent by weight of tocopherols (b).
4. A hair treatment product according to one of claims 1 to 3.
5. A hair treatment product according to of claim 4 useful for protecting the hair against damages by heat drying.
6. A hair treatment product according to of claim 4 useful for protecting the hair against damages by environmental influences and/or hair treatments.
7. The hair treatment product of claim 4, characterized in that the water soluble procyanidins (a) and the oil soluble tocopherols (b) are contained in the aqueous phase of the hair treatment product und have an affinity to the hair.
8. The use of a mixture of (a) water soluble oligomeric procyanidins and (b) oil soluble tocopherols in hair treatment products for protecting further active ingredients against oxidation and/or degradation.
9. The use of a mixture of (a) water soluble oligomeric procyanidins and (b) oil soluble tocopherols in hair treatment products for protecting the skin, particularly the skin of the head, against oxidative stress and/or chemical impairment.

10. A product for care of the mouth according to one of claims 1 to 3.
11. A product for care of the mouth according to claim 10 useful for protecting the gingiva against inflammations and/or parodontosis.
12. A product for care of the mouth according to claim 10 useful for reducing bleeding from the gingiva.
13. A product for care of the mouth according to claim 10 useful for combating plaque and/or caries.
14. The product for care of the mouth according to claim 10, characterized in that the water soluble procyanidins (a) and the oil soluble tocopherols (b) are contained in the aqueous phase of the hair treatment product und have an affinity to the gingiva.
15. The use of a mixture of (a) water soluble oligomeric procyanidins and (b) oil soluble tocopherols in products for care of the mouth for protecting further active ingredients of the product for care of the mouth against oxidation and/or degradation.

Summary

Cosmetic preparations, particularly hair treatment products and products for care of the mouth, having a content of (a) water soluble procyanidins and (b) oil soluble free tocopherols, are described. Such hair treatment products protect the hair, particularly against damage on heat drying and environmental influences, whereas such products for care of the mouth protect the gingiva against parodontosis, reduce bleeding from the gingiva, and combat plaque and caries.